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Simple route to interconnected, hierarchically structured, porous Zn<sub>2</sub>SnO<sub>4</sub> nanospheres as electron transport layer for efficient perovskite solar cellsMeng Zhang<sup>a,c</sup>, Xun Cui<sup>a</sup>, Yufen Wang<sup>a,b,\*\*</sup>, Bing Wang<sup>a</sup>, Meidan Ye<sup>a</sup>, Wenlong Wang<sup>c,\*\*\*</sup>, Chunyuan Ma<sup>c</sup>, Zhiqun Lin<sup>a,\*</sup><sup>a</sup> School of Materials Science and Engineering, Georgia Institute of Technology, Atlanta, GA, 30332, USA<sup>b</sup> Energy & Materials Engineering Centre, College of Physics and Materials Science, Tianjin Normal University, Tianjin, 300387, China<sup>c</sup> National Engineering Laboratory for Reducing Emissions from Coal Combustion, School of Energy and Power Engineering, Shandong University, Jinan, 250061, China

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## ABSTRACT

Constructing electron transport layer (ETL) with higher carrier mobility and suitable bandgap is of key importance as it greatly influences the photovoltaic performance of perovskite solar cells (PSCs). Zn<sub>2</sub>SnO<sub>4</sub> (ZTO) carries a high electron mobility of 10–30 cm<sup>2</sup> V<sup>-1</sup> s<sup>-1</sup>, an order of magnitude over the widely used TiO<sub>2</sub> ETL in PSCs, rendering it an excellent alternative to TiO<sub>2</sub> ETL. Herein, we report a simple yet robust polymer-templating route to interconnected, hierarchically structured, porous ZTO nanospheres as an efficient ETL for high-performance organolead halide PSCs. The porous ZTO nanospheres ETL, composed of an assembly of 4.5-nm ZTO nanoparticles on the surface of porous nanosphere possessing 80–100 nm cavity, renders markedly improved light absorption, enhanced electron extraction, facilitated charger transportation, and suppressed carrier recombination in the resulting PSCs, which exhibit a power conversion efficiency (PCE) of 17.14%, greatly outperforming the device based on the ZTO nanoparticles (14.02%; i.e., without porosity). As such, the strategy for crafting porous yet hierarchically structured semiconductors with high carrier mobility may open up an avenue to create robust ETL, and by extension, hole transport layer (HTL) for high-performance optoelectronics.

## 1. Introduction

The past decade has witnessed tremendous advances in organometal halide perovskite solar cells (PSCs) due to the stellar set of optoelectronic properties of perovskites in conjunction with the low device fabrication cost, facile processability, and easily fine-tuned band gap [1–3]. The electron transport layer (ETL), also functions as a hole blocking layer, represents a key component for high-efficiency PSCs. An ideal ETL material should possess high optical transparency, good electron extraction ability, low carrier transfer resistance and favorable energy level alignment with perovskite [1,4]. Although TiO<sub>2</sub> is the most widely used ETL material, it still presents some electronic and optical shortfalls. It is notable that the electron mobility of mesoporous TiO<sub>2</sub> is less than 1 cm<sup>2</sup> V<sup>-1</sup> s<sup>-1</sup>, much lower than that of CH<sub>3</sub>NH<sub>3</sub>PbX<sub>3</sub> materials (e.g., 10 cm<sup>2</sup> V<sup>-1</sup> s<sup>-1</sup> for CH<sub>3</sub>NH<sub>3</sub>PbI<sub>3</sub>) [5–7]. Moreover, mesoporous TiO<sub>2</sub>-based PSCs are sensitive to ultraviolet (UV) illumination, which

leads to light-induced desorption of surface-adsorbed oxygen, thereby negatively affecting the device stability [8]. Among various alternative ETLs, ZnO and SnO<sub>2</sub> have garnered much attention due to their high electron transport properties and thus high power conversion efficiency (PCE) of the resulting devices. However, ZnO is susceptible to acids and alkali, leading to the decomposition of perovskite in the long term [9, 10]. The SnO<sub>2</sub> ETL suffers from degradation during high-temperature process [11]. More importantly, PSC composed of SnO<sub>2</sub> ETL experiences more severe charge recombination due to the lower conduction band edge of SnO<sub>2</sub> [12]. On the other hand, despite that mesoscopic ZnO<sub>2</sub> and Al<sub>2</sub>O<sub>3</sub> ETLs-based PSCs exhibit good stability in ambient air under AM 1.5 G at 100 mW/cm<sup>2</sup>, their insulating nature results in large resistance for the electron transfer from perovskite absorber to them, consequently reducing short-circuit current density J<sub>sc</sub> [13,14].

Ternary oxide zinc stannate (Zn<sub>2</sub>SnO<sub>4</sub>; ZTO) is a n-type transparent conducting oxide with a high electron mobility of 10–30 cm<sup>2</sup> V<sup>-1</sup> s<sup>-1</sup>, a

\* Corresponding author.

\*\* Corresponding author. School of Materials Science and Engineering, Georgia Institute of Technology, Atlanta, GA, 30332, USA.

\*\*\* Corresponding author.

E-mail addresses: [yfwang@tjnu.edu.cn](mailto:yfwang@tjnu.edu.cn) (Y. Wang), [wwenlong@sdu.edu.cn](mailto:wwenlong@sdu.edu.cn) (W. Wang), [zhiqun.lin@mse.gatech.edu](mailto:zhiqun.lin@mse.gatech.edu) (Z. Lin).

wide optical bandgap of 3.8 eV, and a relatively low refractive index of  $\sim 2.0$  in the visible spectrum [15,16]. More importantly, ZTO is chemically stable with respect to acid, base solution and polar organic solvents [15,16]. Because of its outstanding electrical and optical properties, ZTO has attracted extensive interests for use in gas sensors, Li-ion batteries, dye-sensitized solar cells, and photocatalysis [17–21]. The conduction band edge position of ZTO ( $\sim 4.1$  eV) aligns well with that of  $\text{CH}_3\text{NH}_3\text{PbI}_3$ , rendering it an excellent alternative to  $\text{TiO}_2$  ETL in PSCs. In addition, the wide band gap of ZTO can reduce UV photobleaching and present a higher energy barrier for hole hopping. Nonetheless, the use of ZTO as the ETL for high-efficiency PSCs are comparatively few and limited in scope [16,22–26]. The ability to assemble ZTO nanoparticles into hierarchically structured nanomaterial as ETL may offer expanded flexibility to produce high-performance PSCs. This has yet to be largely explored.

Herein, we report a simple yet robust polymer-templating route to crafting interconnected, hierarchically structured, porous ZTO nanospheres composed of ZTO nanoparticles as the mesoporous electrode (i.e., ETL) for organolead halide PSCs with markedly improved efficiency. Specifically, raspberry-like ZTO nanoparticles are first partially *in-situ* grown on the polystyrene (PS) nanosphere, yielding a patchy core@shell structure (PS@ZTO, i.e., ZTO nanoparticles dispersedly assembled on the surface of PS nanosphere), followed by pyrolysis of PS nanospheres, yielding interconnected, hierarchically structured, porous ZTO nanospheres consisting of ZTO nanoparticles as building blocks. These network-like, hierarchical, porous ZTO nanospheres are employed as a promising ETL for PSCs. Intriguingly, such porous ZTO nanospheres ETL facilitates the infiltration and growth of the perovskite absorber, enhances the light absorption, promotes electron extraction, and accelerates charge transport, as compared to pure ZTO nanoparticles-based devices. Consequently, the PSCs assembled by capitalizing on interconnected yet porous ZTO nanospheres deliver a power conversion efficiency (PCE) of 17.14%, representing an 18% higher than the device using pure ZTO nanoparticles-based ETL (PCE = 14.02%) prepared under the same experimental condition. This study demonstrates that the template-mediated crafting of hierarchical nanostructures may offer a great potential for creating a rich variety of desirable ETL and hole transport layer (HTL) materials with high carrier mobility for applications in optoelectronic devices.

## 2. Experiment Section

### 2.1. Synthesis of interconnected, hierarchically structured, porous ZTO nanospheres (p-ZTO) and solid ZTO nanoparticles (s-ZTO)

An *in-situ* hydrothermal reaction was used to synthesize patchy PS@ZTO nanoparticles core@shell nanostructures, followed by calcination to remove the PS nanospheres, forming interconnected, hierarchically structured, porous ZTO nanospheres composed of ZTO nanoparticles (denoted p-ZTO). Specifically, 1.4024 g  $\text{SnCl}_4 \cdot 5\text{H}_2\text{O}$  and 1.736 g  $\text{Zn}(\text{CH}_3\text{COO})_2 \cdot 2\text{H}_2\text{O}$  were dissolved in a solution containing 16 mL PS spheres emulsion, 32 mL  $\text{CH}_2(\text{OH})\text{CH}(\text{OH})\text{CH}_3$ , 16 mL deionized  $\text{H}_2\text{O}$ , and 16 mL  $\text{HO}(\text{CH}_2)_2\text{NH}(\text{CH}_2)_2\text{OH}$ . The size of the PS nanosphere template is  $\sim 100$  nm. After stirring for 30 min, the mixture was placed into a stainless-steel autoclave with a Teflon inner layer. The reaction was performed at 200 °C for 24 h, and the resulting PS@ZTO nanoparticles was separated by filtration and washed with deionized water and ethanol for three times each. Finally, the sample was dried in oven at 70 °C. Then, PS@ZTO nanoparticles was dispersed in ethanol and spin-coated onto the compact ZTO-coated FTO glass, yielding p-ZTO film on its surface. Subsequent pyrolysis to remove PS nanospheres was conducted at 500 °C for 1 h in air with a ramping rate of 2 °C/min, leaving behind interconnected, hierarchically structured, porous ZTO nanospheres composed of ZTO nanoparticles.

Solid ZTO nanoparticles were also prepared with the same method yet without the addition of the PS nanospheres as template (denoted s-

ZTO). The ZTO nanoparticles film was obtained and annealed in the same way as that of p-ZTO film.

### 2.2. Solar cell fabrication

Fluorine-doped tin oxide ( $\text{F-SnO}_2$ )-coated transparent conduction glass substrates (FTO) were etched using metallic Zn powder and HCl (4 M), then cleaned with detergent, DI water, acetone, isopropanol, and ethanol under an ultrasonic condition for 30 min, respectively, and lastly purged with nitrogen. The substrates underwent an oxygen plasma treatment for 15 min prior to the deposition of the compact ZTO hole blocking layer. The compact ZTO layer was deposited by spin-coating a solution containing  $\text{ZnCl}_2$  and  $\text{SnCl}_2$  (Zn/Sn ratio = 2) at 3000 rpm for 30 s, followed by annealing at 500 °C for 1 h. The mesoporous ZTO film was deposited on the compact ZTO-coated FTO glass by spin-coating the ZTO nanoparticles solution prepared with or without the use of PS templates at 2000 rpm for 30 s. After drying at room temperature, the samples were annealed at 500 °C for 1 h. The deposition of the  $\text{CH}_3\text{NH}_3\text{PbI}_3$  absorber layer and spiro-OMeTAD HTL were carried out in a glove box. The  $\text{CH}_3\text{NH}_3\text{PbI}_3$  film was formed using a one-step spin-coating procedure. The  $\text{CH}_3\text{NH}_3\text{PbI}_3$  precursor solution was prepared by dissolving 461 mg of  $\text{PbI}_2$  and 159 mg of  $\text{CH}_3\text{CH}_2\text{I}$  in a mixed solution containing 0.64 mL of N,N-dimethylformamide and 71  $\mu\text{L}$  of dimethylsulfoxide, with stirring for 4 h at 70 °C. The as-prepared precursor solution was spun on the mesoporous ZTO film (i.e., p-ZTO or s-ZTO) at 4000 rpm for 25 s using diethyl ether as anti-solvent. After spinning, the film was dried at 65 °C for 5 min and then 100 °C for 10 min to remove the residual solvent. The spiro-OMeTAD-based HTL solution was prepared by adding 72.3 mg of spiro-OMeTAD, 28.8  $\mu\text{L}$  of 4-*tert*-butylpyridine and 17.5  $\mu\text{L}$  of Li-bis-(tri-fluoromethanesulfonyl) imide (Li-TFSI) solution (520 mg of Li-TFSI in 1 mL of acetonitrile) in 1 mL of chlorobenzene. The resulting solution was deposited onto the perovskite-coated substrate by spin-coating at 3000 rpm for 30 s. Finally, 120 nm of silver was deposited through thermal evaporation as the counter electrode.

### 2.3. Characterization

The crystalline structures of s-ZTO nanoparticles and p-ZTO nanospheres were examined using X-ray diffraction (X'pert PRO, Netherlands) using Cu K $\alpha$ 1 ( $\lambda = 1.5401$  Å) radiation. The surface morphologies of the mesoporous ZTO layers and the perovskite films were characterized by field-emission scanning electron microscopy (SEM, Hitachi SU8000, Japan) equipped with high energy electron sources and stunningly sensitive detector system. Transmission electron microscopy (TEM) imaging was conducted using a JME-2100 TEM. UV–vis light absorption spectra were recorded with a UV–vis spectrophotometer (SHIMADZU, UV-2600) with Halogen lamp and Deuterium lamp as excitation sources by using  $\text{BaSO}_4$  standard plate as reference. The steady-state photo-luminescence spectra were obtained using a Spectrofluorophotometer (RF-5301PC, SHIMADZU) with the excitation wavelength of 580 nm. The time-resolved photoluminescence measurements were performed using a Photon Technology International (PTI) LaserStrobe Spectrofluorometer equipped with a PTI GL-3300 ns nitrogen laser ( $\lambda = 337$  nm) and photomultiplier tubes (PMT) together with time-correlated single photon counting (TCSPC) for single collection and analysis.

For the device characterization, the current density-voltage (*J-V*) characteristics and the steady-state photocurrent were measured using a Newport AM 1.5G solar simulator together with a Keithley 2601A multisource meter under illumination of 100  $\text{mW}/\text{cm}^2$ . The external quantum efficiency (EQE) were examined as a function of wavelength from 300 nm to 800 nm using a Newport Quantum Efficiency Measurement Kit. The internal impedance of the devices was obtained using electrochemical impedance spectroscopy (EIS) using Zennium electrochemical workstation (Zahner) with the frequency ranging from 0.01 Hz

to 100 kHz at room temperature under dark condition with an applied bias voltage. The controlled intensity modulated photovoltage spectroscopy (CIMVS) and controlled intensity modulated photocurrent spectroscopy (CIMPS) measurements were carried out using the Zahner electrochemical workstation with a light source control module.

### 3. Results and discussion

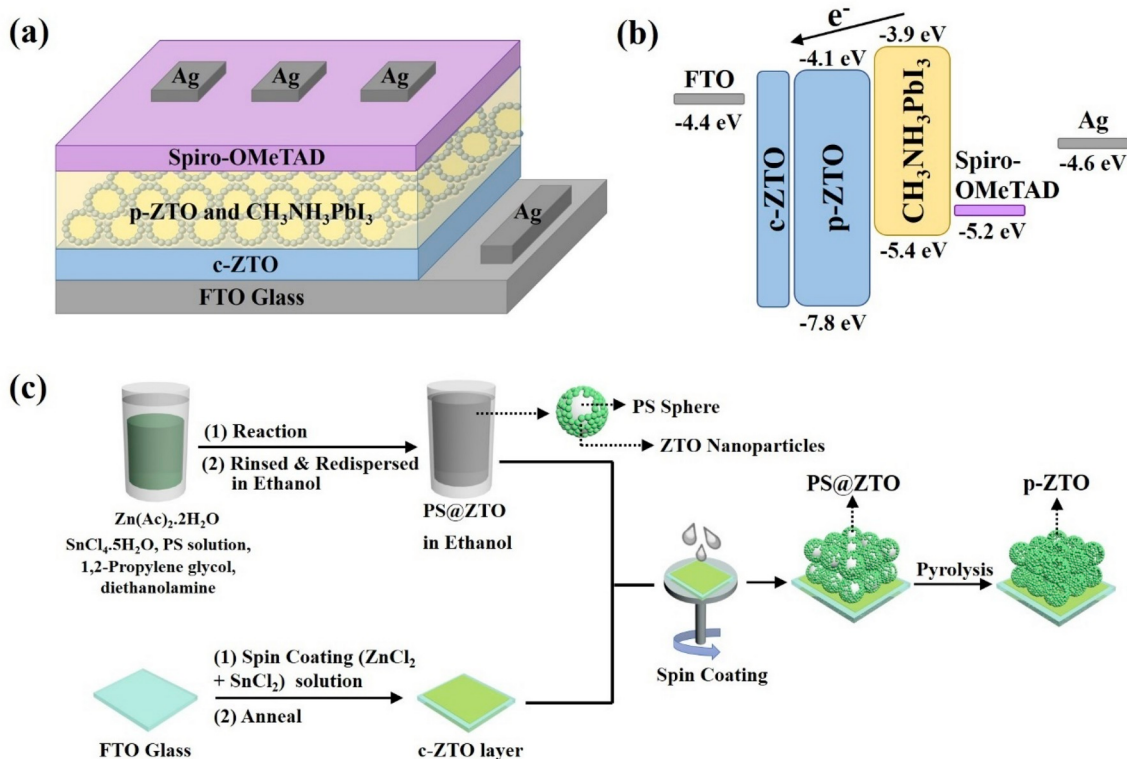
The device configuration and the energy level alignment of all constituents in the PSC are shown in Fig. 1a–b, respectively. Notably, the ETL is composed of a thin compact ZTO layer and an interconnected, hierarchically structured, porous ZTO nanospheres. The compact ZTO film (denoted c-ZTO), also served as a hole-blocking layer, was prepared by spin-coating a solution containing  $\text{ZnCl}_2$  and  $\text{SnCl}_2$ , followed by annealing (see *Experiment Section*). Subsequently, the interconnected porous ZTO nanospheres was deposited by spin-coating the solution of PS@ZTO nanoparticles on the surface of c-ZTO film, followed by pyrolysis to remove the PS nanospheres (Fig. 1c).

To prepare the PS@ZTO nanoparticles solution dissolve in ethanol, the ZTO precursors (i.e.,  $\text{SnCl}_4 \cdot 5\text{H}_2\text{O}$  and  $\text{Zn}(\text{CH}_3\text{COO})_2 \cdot 2\text{H}_2\text{O}$ ), 100-nm PS nanospheres,  $\text{CH}_2(\text{OH})\text{CH}(\text{OH})\text{CH}_3$ , deionized  $\text{H}_2\text{O}$ , and HO  $(\text{CH}_2)_2\text{NH}(\text{CH}_2)_2\text{OH}$  were introduced in an autoclave. Interestingly, the hydrothermal reaction yielded raspberry-like 4-nm ZTO nanoparticles partially covered on the surface of PS nanosphere (Fig. S1), forming a patchy core@shell structure. The subsequent calcination effectively removed the PS template, leaving behind interconnected, hierarchically structured, porous ZTO nanospheres comprising ZTO nanoparticles (right panel in Fig. 1c). It is important to note the incomplete surface coverage of ZTO nanoparticles on the PS template, in conjunction with the use of PS nanosphere template that effectively prevented ZTO nanoparticles themselves from aggregation, attributed to the attained interconnected porous ZTO nanospheres, thereby facilitating the infiltration of the perovskite absorber as discussed later. Such network-like

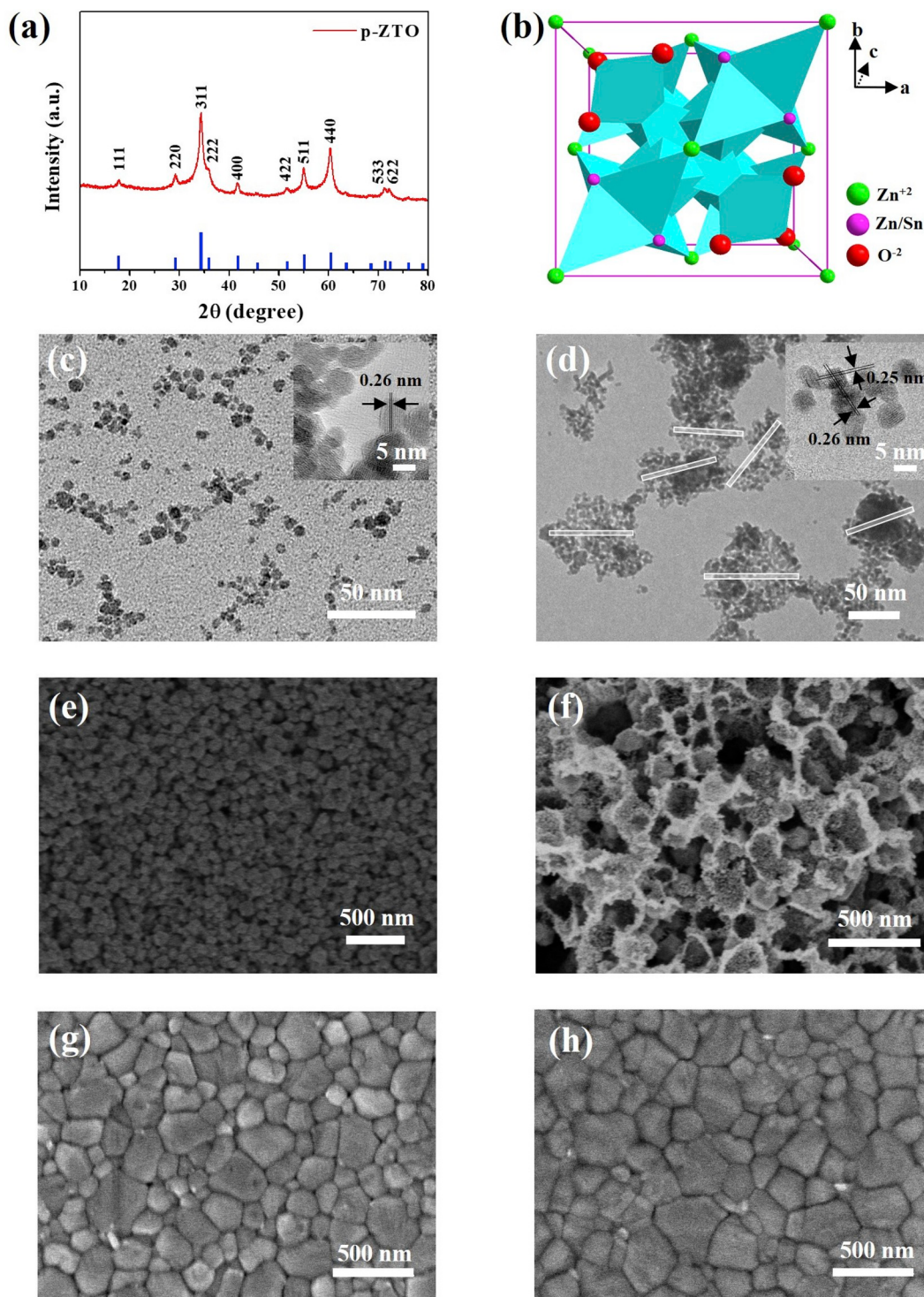
yet porous nanostructures are advantageous for them to be used as mesoporous ETL for PSCs. For comparison, the control devices with solid ZTO nanoparticles-based framework were also fabricated, where the ZTO nanoparticles were synthesized using the same hydrothermal procedure yet without the addition of PS template (see *Experiment Section*).

Subsequently, the homogeneous  $\text{CH}_3\text{NH}_3\text{PbI}_3$  layer was deposited as an absorber layer on the ZTO ETL, followed by spin-coating of spiro-OMeTAD as a HTL and thermal evaporation of current-collecting Ag anode (see *Experiment Section*). A representative cross-sectional SEM image of the resulting PSC is shown in Fig. S2a. As schematically illustrated in Fig. 1b, the energy levels of ZTO ETL,  $\text{CH}_3\text{NH}_3\text{PbI}_3$  perovskite absorber, and spiro-OMeTAD HTL, suggesting the favorable energy alignment at the ETL/perovskite and perovskite/HTL interfaces for efficient charge separation and transport, which is beneficial for maximizing the built-in voltage of the fabricated device to yield optimum open-circuit voltage ( $V_{oc}$ ). The conduction band edge of ZTO is similar to that of widely used  $\text{TiO}_2$ , implying its similar working mechanism to a  $\text{TiO}_2$ -based PSC. However, the wider bandgap and faster electron mobility of ZTO make it a more compelling electron transport material over  $\text{TiO}_2$ .

Hereafter, interconnected, hierarchically structured, porous ZTO nanospheres and ZTO nanoparticles synthesized in the absence of the PS template (i.e., control sample) are referred to as *porous ZTO nanospheres* (p-ZTO nanospheres) and *solid ZTO nanoparticles* (s-ZTO nanoparticles), respectively. The structure and phase composition of as-prepared p-ZTO was first characterized by powder X-ray diffraction (XRD; Fig. 2a). The sharp peaks were found at the same diffraction positions both for p-ZTO nanospheres and s-ZTO nanoparticles (Fig. S2b), indicating that the composition and crystalline structure of ZTO did not change during the incorporation and removal of PS template. However, better crystallization was found for p-ZTO as compared to s-ZTO due to the pyrolysis of the former. Diffraction peaks can be indexed as the (111), (220), (311), (222), (400), (422), (511), (440), (533), and (622) reflections,



**Fig. 1.** (a) Schematic 3D view of the perovskite solar cell (PSC). (b) Energy level diagram of components in the PSC based on the  $\text{Zn}_2\text{SnO}_4$  (ZTO) ETL. (c) Schematic illustration of synthesis of hollow ZTO nanospheres partially decorated on the PS nanosphere surface (upper left panels) and their subsequent deposition and processing (i.e., pyrolysis of PS nanospheres) to yield interconnected, hierarchically structured, porous ZTO nanospheres comprising ZTO nanoparticles.



**Fig. 2.** (a) The XRD pattern of interconnected, hierarchically structured, porous  $\text{Zn}_2\text{SnO}_4$  (ZTO) nanospheres (i.e., p-ZTO nanospheres). The peaks are assigned to cubic ZTO according to JCPDS No. 21–1272 (bottom panel). (b) The structure of a cubic ZTO unit cell. TEM images of (c) ZTO nanoparticles (i.e., s-ZTO nanoparticles) and (d) p-ZTO. Inset in (c–d) are the HRTEM images. FE-SEM images of (e) s-ZTO nanoparticles film and (f) p-ZTO nanospheres film on the FTO glass. SEM images of the perovskite layer spin-coated on the surface of (g) s-ZTO nanoparticles film and (h) p-ZTO nanospheres film.

respectively, corresponding to the cubic structure of ZTO (JCPDS Card No. 24–1470) with cell constants of  $a = b = c = 8.657 \text{ \AA}$ . Moreover, no peaks from impurity were observed in these samples. Based on the Scherrer's equation [27], the average size of ZTO nanoparticle in p-ZTO and in s-ZTO are approximately 4.3 nm and 6.7 nm, respectively, from the full width at half maximum of the (311) peak. Fig. 2b depicts the structure of a cubic-type spinel ZTO unit cell. The typical transmission

electron microscopy (TEM) images of as-synthesized s-ZTO nanoparticles and p-ZTO nanospheres are shown in Fig. 2c and d, respectively. The low-magnification TEM image shows the highly dispersed ZTO nanoparticles with an average size of approximately 6.9 nm (Fig. 2c and Fig. S3a), in accordance with the XRD results. The high-resolution TEM images (insets in Fig. 2c–d) shows that the ZTO nanoparticles have clear lattice fringes, suggesting their high crystallinity. Fig. 2d

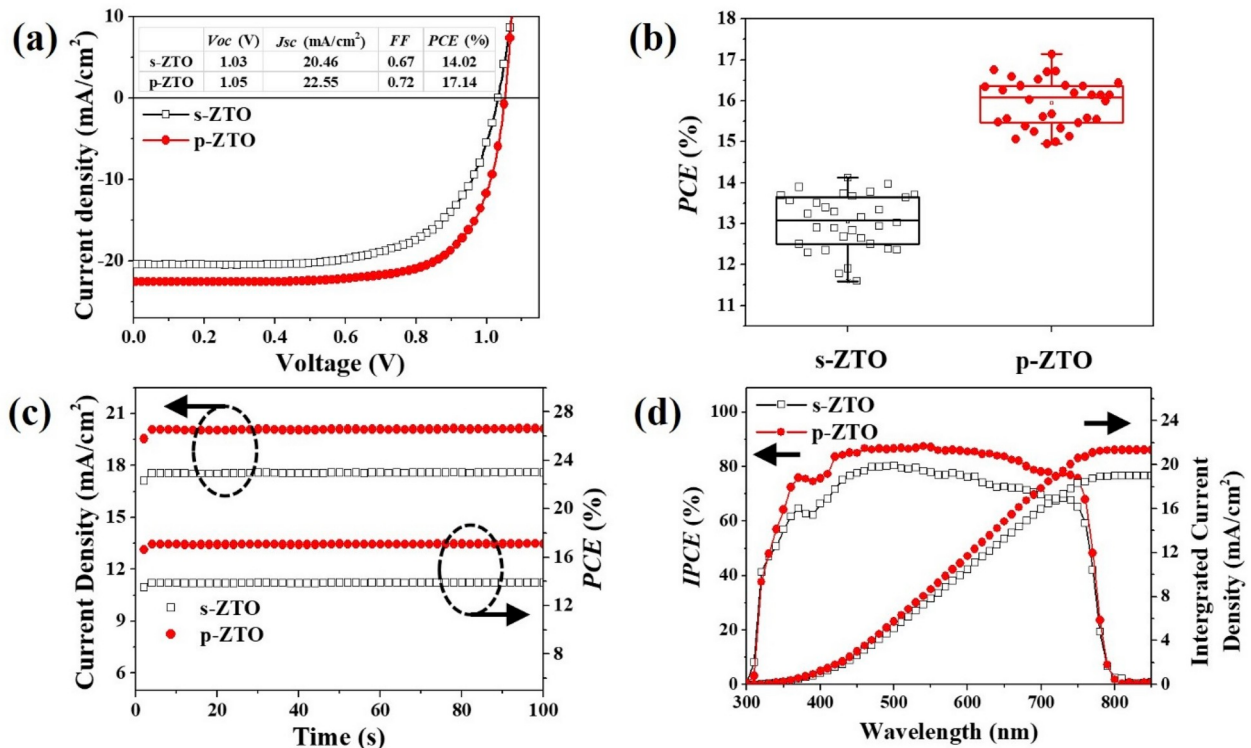
presents the TEM image of p-ZTO, where the morphology of nanoparticles retains, compared to the control sample (Fig. S3a), yet with a smaller and more uniform size of 4.5 nm (Fig. S3b). The 0.26-nm and 0.25-nm lattice spacing in HRTEM images can be assigned to the ZTO (311) and (222) planes, respectively (insets in Fig. 2c–d). The decreased nanoparticle size in p-ZTO sample is due likely to the prevention of ZTO nanoparticle aggregation enabled by the use of PS template during the in-situ ZTO nanoparticle growth. The size of p-ZTO nanospheres is ~80–100 nm, which is slightly decreased compared to the size of the PS nanosphere template (~100 nm). This can be ascribed to the thermal shrinkage during the calcination.

The mesoporous structures of ETL material has a great effect on the quality and surface morphology of the perovskite layer deposited, which in turn significantly affects the device performance. To eliminate the effect of ZTO crystallization on the corresponding devices, s-ZTO nanoparticles film was annealed in the same way as that of p-ZTO nanospheres film. Clearly, the p-ZTO nanospheres display a more prominent porous and hierarchical structure (Fig. 2f) than that of s-ZTO nanoparticles (control sample; Fig. 2e). The s-ZTO nanoparticles film has rather uniform morphology and porosity, yet the nanoparticles severely agglomerated with an average size of approximately 100 nm due to the spin-coating and annealing process. In contrast, the p-ZTO nanospheres film not only demonstrates a higher porosity with open structure, but also retains the original uniform size of nanoparticle building blocks owing to the protection of the PS template. Thus, the large porosity, high surface area, and hierarchical structure of p-ZTO film over the s-ZTO nanoparticles film resulted in the better perovskite absorber infiltration and growth, as revealed by SEM measurement, where a compact and homogeneous surface morphology without microscopic pinholes is clearly evident (Fig. 2h). The densely packed grain size is approximately 200–400 nm, larger than that of the perovskite film grown on the s-ZTO nanoparticles film (Fig. 2g). This in turn renders the improved charge separation and transport due to the reduced grain boundaries and suppressed charge recombination [28].

A markedly improved photovoltaic performance was found for the p-ZTO-nanospheres-incorporated devices over that of s-ZTO-nanoparticles-based devices, as shown in Fig. 3a. The former devices (p-ZTO-based) delivered a short-circuit photocurrent density  $J_{sc}$  of 22.55 mA/cm<sup>2</sup>, an open-circuit voltage  $V_{oc}$  of 1.05 V, and a fill factor  $FF$  of 0.72, leading to a  $PCE$  of 17.14%. This contrasts the latter device (s-ZTO-based) with a lower  $J_{sc}$  of 20.46 mA/cm<sup>2</sup>,  $V_{oc}$  of 1.03 V,  $FF$  of 0.67 and a  $PCE$  of 14.02%. Fig. S4 and Fig. 3b summarizes the statistics of photovoltaic parameters extracted from the  $J$ - $V$  curves of 32 independent devices. The  $PCE$ s were reproducible for both s-ZTO nanoparticles and p-ZTO nanosphere-based PSCs. The average  $PCE$ s of  $15.86 \pm 0.68\%$  and  $12.16 \pm 0.65\%$  were attained for p-ZTO and s-ZTO-based devices, respectively. The stable photocurrent measurement was conducted for both devices at their maximum  $PCE$  under the standard illumination (AM 1.5G) (Fig. 3c). A stable output power corresponding to a  $PCE$  of 17.06% and 13.88% were found for p-ZTO nanospheres and s-ZTO nanoparticles-based champion devices, respectively, in good agreement with the  $J$ - $V$  measurements.

Fig. 3d presents the corresponding incident-photon-to-current conversion efficiency ( $IPCE$ ) spectra of the devices. All devices possess a broad  $IPCE$  plateau (over 70%) over 400–760 nm, suggesting the superior photo-response of the perovskite absorber. However, a notable improvement was seen for p-ZTO-incorporated devices over s-ZTO-based device, signifying a lower recombination loss in p-ZTO-incorporated devices [25]. The estimated current density by integrating the  $IPCE$  spectrum with the AM 1.5G solar spectrum (Fig. 3d) correlated well with those obtained from the  $J$ - $V$  curves. As  $IPCE = LHE \times \phi_{inj} \times \eta_{cc}$ , where  $LHE$  is the light harvesting efficiency,  $\phi_{inj}$  is the electron-injection efficiency, and  $\eta_{cc}$  is the charge-collection efficiency [29]. Nonetheless, we note that the improved performance of the p-ZTO-based devices lies in not only the greater light absorption of the perovskite layer as noted above, but also enhanced electron extraction, facilitated charge transport, and suppressed carrier recombination, as detailed below.

Fig. 4a shows the UV-vis absorption spectra of CH<sub>3</sub>NH<sub>3</sub>PbI<sub>3</sub> films



**Fig. 3.** (a)  $J$ - $V$  curves and (b) photovoltaic performance statistics of PSCs by exploiting p-ZTO nanospheres and s-ZTO nanoparticles, respectively, as ETLs under illumination of 100 mW/cm<sup>2</sup> (AM 1.5 G). (c) The stable  $J_{sc}$  and  $PCE$  of PSCs measured near the maximum  $PCE$ , and (d) its corresponding  $IPCE$  spectrum (left y axis) and integrated photocurrent density (right y axis).

deposited on the two different ZTO-based layers. A slightly higher absorption was observed for the  $\text{CH}_3\text{NH}_3\text{PbI}_3$  film spun on the p-ZTO nanospheres film over that of the  $\text{CH}_3\text{NH}_3\text{PbI}_3$  film coated on s-ZTO nanoparticles film, which contributes to the enhanced  $J_{sc}$  and  $IPCE$ . The relatively high absorption for the former is due largely to the interconnected porous structures of p-ZTO nanospheres, which imparts the better infiltration and growth of the perovskite absorber, thus enhancing its light absorption. Moreover, the high porosity of p-ZTO nanospheres leads to relatively high transparency, which effectively avoids the parasitic absorption of the ZTO ETL and thus maximize the resulting photocurrent of PSCs.

To evaluate the charge transfer dynamics from the perovskite layer to the ZTO ETL, photoluminescence (PL) and time-resolved PL (TRPL) measurements on the  $\text{CH}_3\text{NH}_3\text{PbI}_3$  film deposited on the two different ZTO layers were performed (Fig. 4b–c). Compared to the pristine  $\text{CH}_3\text{NH}_3\text{PbI}_3$ , the decreased PL intensity was due to the effective charge transfer from perovskite to ZTO. A greater emission reduction of the perovskite layer on the p-ZTO layer (a 85.4% quenching) than that on the s-ZTO paper (a 76.5% quenching) suggests an enhanced charge extraction at the p-ZTO/ $\text{CH}_3\text{NH}_3\text{PbI}_3$  interface. The fitting of the TRPL data with bi-exponential decay yields a fast decay  $\tau_1$  and a slow decay  $\tau_2$ , corresponding to the intrinsic band-to-band recombination and trap-assisted recombination, respectively [30]. The charge-carrier lifetime decreased sharply in the perovskite films on the p-ZTO surface ( $\tau_1 = 5.11$  ns;  $\tau_2 = 19.02$  ns) compared to that on the s-ZTO surface ( $\tau_1 = 6.83$  ns;  $\tau_2 = 43.68$  ns), signifying the more efficient photo-generated electron extraction for  $\text{CH}_3\text{NH}_3\text{PbI}_3$  deposited on p-ZTO, which is in good agreement with the steady-state PL. The enhanced charge extraction with p-ZTO film can be attributed to the improved contact due to better perovskite infiltration and the abundant transfer pathways provided by the interconnected, hierarchically structured, porous p-ZTO film. On the other hand, improved perovskite growth on p-ZTO film results in higher-quality perovskite crystallization with larger perovskite grains and less grain boundaries, as corroborated by SEM (Fig. 2h), thereby reducing trap sites at the p-ZTO/ $\text{CH}_3\text{NH}_3\text{PbI}_3$  interface. The decreased traps further suppress carrier recombination [28].

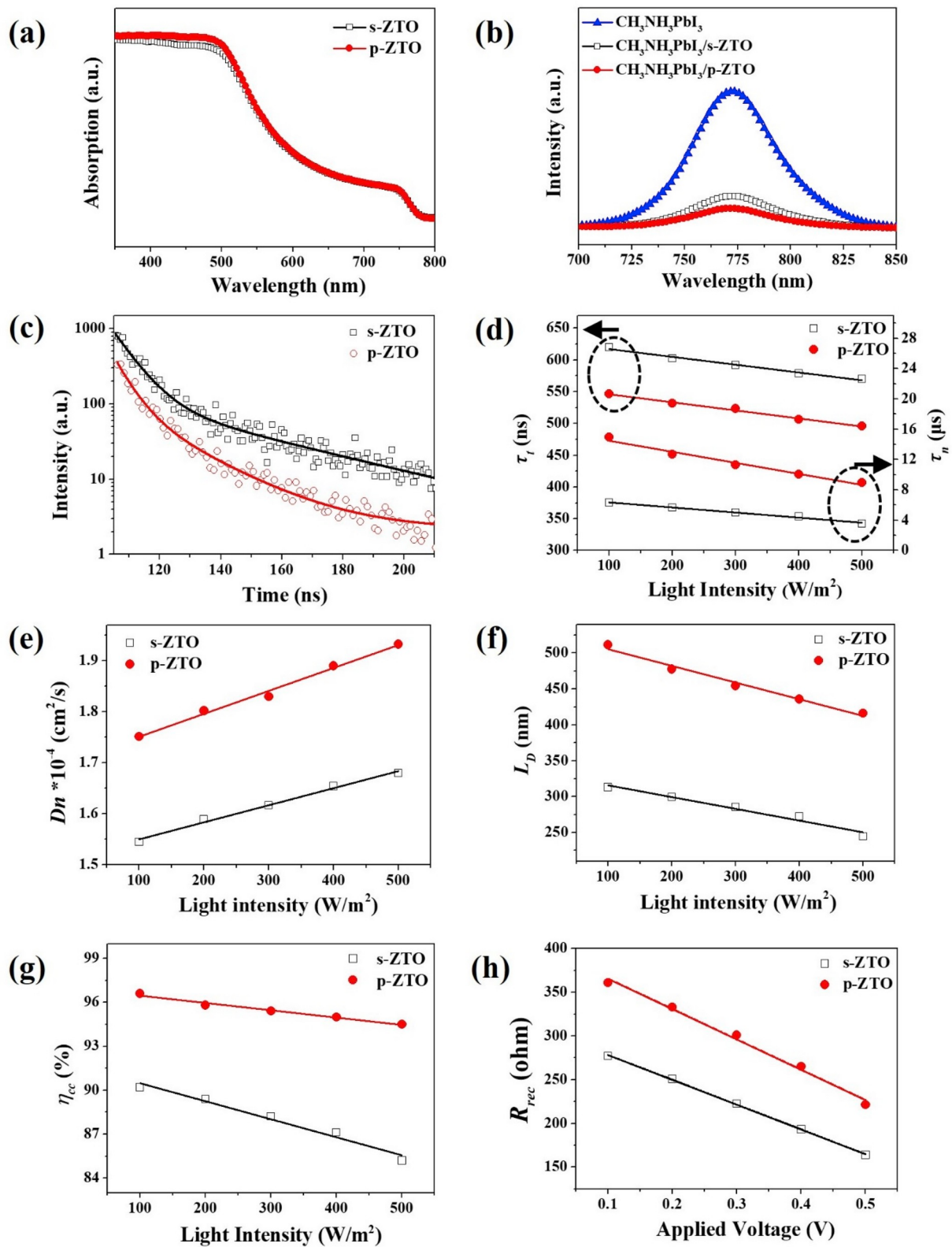
The charge transport dynamics along the entire pathway in devices with the two different ZTO layers incorporated were scrutinized by the controlled intensity modulated photocurrent spectroscopy (CIMPS) and controlled intensity modulated photovoltage spectroscopy (CIMVS) measurements. A shorter carrier transition time  $\tau_t$  and a longer carrier lifetime  $\tau_n$  were found for the p-ZTO-based devices than that of the s-ZTO-based devices (Fig. 4d). The carrier transition time is calculated according to the following equation  $\tau_t = 1/(2\pi f_{t,min})$ , where  $f_{t,min}$  is the characteristic frequency at the minimum of the CIMPS imaginary component [31]. On the other hand, the carrier lifetime is obtained using the equation  $\tau_n = 1/(2\pi f_{n,min})$ , where  $f_{n,min}$  is the corresponding frequency when the imaginary part of photovoltage reaches the minimum value [31]. The representative Nyquist plots and Bode plots of CIMPS and CIMVS characterizations are shown in Figs. S5 and S6, respectively. The shorter  $\tau_t$  indicates a faster carrier transport, which can be attributed to the improved contact between p-ZTO and  $\text{CH}_3\text{NH}_3\text{PbI}_3$  and ample transfer pathways rendered by the network-like structure of p-ZTO as noted above. A facilitated charge transport with the p-ZTO incorporation mitigates the recombination process, thus extending  $\tau_n$ . The electron diffusion coefficient  $D_n$  can be calculated based on the equation  $D_n = \omega^2/(2.35\tau_t)$ , where  $\omega$  is the thickness of photoanode ( $\omega = 150$  nm; estimated from the cross-sectional SEM image of the device (Fig. S2a)). As shown in Fig. 4e,  $D_n$  values for the p-ZTO-based devices are much higher than that of the s-ZTO-based devices, suggesting that the photogenerated electrons can diffuse more rapidly from perovskite to the FTO substrate, and thus effectively reduce the charge recombination during the charge transport. With the  $D_n$  and  $\tau_n$ , the electron diffusion length  $L_D$  can be obtained according to  $L_D = (D_n * \tau_n)^{1/2}$ . The  $L_D$  of the devices based on p-ZTO nanospheres was found to increase by approximately 1.5 folds, compared to that based on s-ZTO nanoparticles

(Fig. 4e). The longer  $\tau_n$  and decreased  $\tau_t$  also result in a higher charge collection efficiency  $\eta_{cc}$  ( $\eta_{cc} = 1 - (\tau_t/\tau_n)$ ). As displayed in Fig. 4g, the p-ZTO-based device possesses a  $\eta_{cc}$  between 96.6 and 94.5% as a function of light intensity, while the s-ZTO-based device has a  $\eta_{cc}$  between 90.2 and 85.2%. The increased  $\eta_{cc}$  for the former device is a result of the enhanced electron extraction and promoted charge transport, as discussed above.

The electrochemical impedance spectroscopy (EIS) measurements were further conducted to elucidate carrier recombination and interfacial charge-transport process with applied bias from 0.1 to 0.6 V under standard AM 1.5 illumination (Fig. S7). The Nyquist plots were fitted to an equivalent circuit, consisting of a series resistance  $R_s$  and an inner recombination resistance  $R_{rec}$  (Fig. S7c) [32,33]. It is notable that the  $R_{rec}$  is dominant in both p-ZTO nanospheres-based and s-ZTO nanoparticles-based devices. However, the p-ZTO nanospheres-based devices demonstrate a much larger  $R_{rec}$  than that of s-ZTO nanoparticles-based devices (Fig. 4h). A larger  $R_{rec}$  in the p-ZTO nanospheres-based devices relates to a lower charge recombination and signifies more efficient charge separation and transport, which is a direct consequence of the promoted charge transfer due to hierarchical structure of p-ZTO and alleviated p-ZTO/perovskite interfacial charge loss because of improved perovskite crystallization, correlating well with the studies described above. Taken together, the higher device performance was resulted in.

The hysteresis of the two devices (i.e., p-ZTO nanospheres-based and s-ZTO nanoparticles-based) was examined in the forward and reverse scan modes. Fig. 5a compares their typical  $J$ - $V$  curves, where extracted  $J_{sc}$ ,  $V_{oc}$  and  $FF$  parameters are summarized as an inset. For the p-ZTO-based devices, the  $PCE$ s are 17.14% and 16.25% for the reverse and forward scans, showing a 5.19% difference in  $PCE$  and thus limited hysteresis. In contrast, s-ZTO-based devices exhibit rather serious hysteresis with a 10.56%  $PCE$  difference. It is widely recognized that the hysteresis is originated from the structural defects within the perovskite layer [34]. Notably, the defects of the perovskite absorber and the associated interfaces act as traps for electrons and holes, contributing to the hysteresis [34,35]. As evidence by SEM image (Fig. 2f), the perovskite film on the p-ZTO layer possesses large grains and reduced grain boundaries, which in turn leads to decreased interfacial trap states between ZTO ETL and  $\text{CH}_3\text{NH}_3\text{PbI}_3$ . On the other hand, interconnected, hierarchically structured and porous characteristics of p-ZTO nanospheres imparts the rapid charge extraction and transport, facilitates quick dissipation of capacitive charges, thus reduces the device hysteresis.

Long-term stability of PSCs is of key importance for practical applications. The  $J$ - $V$  characteristics of the p-ZTO-based and s-ZTO-based devices without encapsulation were monitored under dark storage in dry air for one month. The corresponding  $PCE$  change was summarized in Fig. 5b. Clearly, good stability was found for devices. The results show that the  $PCE$  marginally increased during the first several days stability test, and then insignificantly decreased mainly due to slightly decreased  $J_{sc}$ . Specifically, the p-ZTO-based devices retained a  $PCE$  of 15.43% with a drop of 10% of its initial value, while the s-ZTO-based devices maintained 85% of its initial efficiency after 30 days. The slight  $PCE$  improvement in the first few days can be attributed to the enhanced p-type conductivity in the LiTFSI-doped spiro-MeOTAD after storage in dry air [36]. The subsequent minor decrease of device efficiency may be related to the decomposition of  $\text{CH}_3\text{NH}_3\text{PbI}_3$  [37]. It is worth noting that the excellent stability of the ZTO ETL-based PSCs may be due to the strong interfacial bonding between the perovskite and the ZTO layers, which in turn prevents the moisture penetration [24,38]. The comparatively better stability of the p-ZTO-based devices could be credited to the improved morphology of the perovskite layer. As grain boundary and structural defect are the sites accessible to moisture [38], the perovskite layer situated on the p-ZTO nanosphere film features large grain size and reduced grain boundary, thereby increasing the stability of the resulting devices.



**Fig. 4.** (a) Absorption, (b) photoluminescence, and (c) time-resolved photoluminescence spectra of  $\text{CH}_3\text{NH}_3\text{PbI}_3$  films deposited on p-ZTO nanospheres film and s-ZTO nanoparticles film, respectively. (d) Carrier transition time  $\tau_t$  and carrier lifetime  $\tau_n$ , (e) electron diffusion coefficient  $D_n$ , (f) electron diffusion length  $L_D$ , and (g) charge collection efficiency  $\eta_{cc}$  of devices, based on p-ZTO nanospheres and s-ZTO nanoparticles as ETLs, as a function of incident light intensity derived from the CIMPS/CIMVS measurements. (h) Recombination resistance  $R_{rec}$  of devices, based on p-ZTO nanospheres and s-ZTO nanoparticles as ETLs, derived from the electrochemical impedance spectroscopy as a function of applied bias under AM1.5 sunlight.

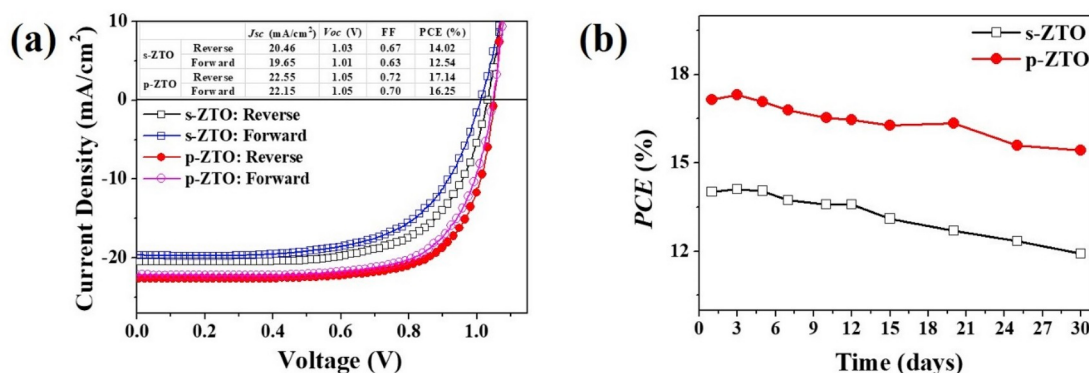


Fig. 5. (a) Hysteresis analysis, and (b) stability test of p-ZTO nanospheres-based and s-ZTO nanoparticles-based devices.

## 4. Conclusions

In summary, we demonstrated a simple yet viable polymer-templating means of producing interlaced porous ZTO nanospheres of tens of nanometers comprising a few nanometer-sized ZTO nanoparticles (i.e., a hierarchical structure; p-ZTO) as the ETL for high-efficiency organolead halide PSCs. The resulting p-ZTO carries high porosity and large surface area that promotes the infiltration and growth of perovskite absorber, resulting in improved perovskite crystallization and large grain sizes as well as increased light adsorption. More importantly, the effective perovskite infiltration and sufficient charge transfer routes enabled by the interconnected porous p-ZTO collectively enhance electron extraction and facilitate charge transport. Moreover, fast charge transfer and good perovskite crystallization account for the limited hysteresis. The p-ZTO-based device retains a 90% of its original PCE over a period of one month without encapsulation, manifesting its outstanding stability. Clearly, as an n-type high carrier mobility semiconductor, ZTO exemplifies an attractive alternative to the commonly used TiO<sub>2</sub> as highly effective ETL. By extension, the polymer-templating strategy affords convenient access to a wide diversity of semiconductor nanostructures of interest with interconnected yet porous attributes as robust electron or hole transport materials for applications in areas such as solar cells, photodetectors, sensors. etc.

## Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

## CRediT authorship contribution statement

**Meng Zhang:** Writing - original draft. **Xun Cui:** Formal analysis. **Yufen Wang:** Writing - original draft. **Bing Wang:** Formal analysis. **Meidan Ye:** Formal analysis. **Wenlong Wang:** Writing - review & editing. **Chunyu Ma:** Writing - review & editing. **Zhiqun Lin:** Writing - original draft.

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## Appendix A. Supplementary data

Supplementary data to this article can be found online at <https://doi.org/10.1016/j.nanoen.2020.104620>.

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**Dr. Meidan Ye** is an associate Professor in the Research Institute for Soft Matter and Biomimetics, Department of Physics, College of Physical Science and Technology at Xiamen University. She received her PhD in Physical Chemistry from the State Key Laboratory of Physical Chemistry of Solid Surfaces, and College of Chemistry and Chemical Engineering at Xiamen University in 2014. Her research interests include advanced nanostructured metal oxide/sulfide materials for energy and environment applications, such as perovskite/dye/quantum dot-sensitized solar cells, photocatalysis, electrochemical energy storage devices and wearable sensors.



**Dr. Wenlong Wang** is a professor in the School of Energy and Power Engineering at Shandong University, China, and the director of Shandong Provincial Engineering Lab for Solid Waste Green Materials. He received his Ph.D. degree in Power Engineering and Engineering Thermodynamics from Zhejiang University, China in 2002. His research interests include solid waste management and utilization, green cementitious materials, green carbonaceous materials, gas pollutants control, microwave heating and pyrolysis, CO<sub>2</sub> capture and fixation.



**Dr. Chunyuan Ma** is a professor in the School of Energy and Power Engineering at Shandong University, China. He received his Ph.D. degree in Thermal Power Engineering from Harbin Institute of Technology, China in 1996. His research interests include clean energy production, air pollution control and advanced energy materials.



**Dr. Zhiqun Lin** is a Professor in the School of Materials Science and Engineering at the Georgia Institute of Technology. He received his Ph.D. degree in Polymer Science and Engineering from the University of Massachusetts, Amherst in 2002. His research interests include electrocatalysis, batteries, photocatalysis, solar cells, LEDs, semiconductor organic-inorganic nanocomposites, quantum dots (rods), multifunctional nanocrystals, and Janus nanostructures, conjugated polymers, block copolymers, polymer blends, hierarchical structure formation and assembly, and surface and interfacial properties.



**Dr. Meng Zhang** received her Ph.D. degree in Engineering Thermophysics from Zhejiang University, China in 2016 and then worked as a research assistant at the Shandong University. She joined Dr. Zhiqun Lin's group in the School of Materials Science and Engineering at the Georgia Institute of Technology as a postdoc research fellow in Aug. 2017. Her research interests are centered on energy conversion and storage, including perovskite solar fuels, CO<sub>2</sub> conversion and utilization, and advanced energy materials.



**Dr. Xun Cui** received his Ph.D. degree in Chemical Engineering and Technology from Chongqing University in 2018. Now he is a postdoctoral fellow in the School of Materials Science and Engineering at the Georgia Institute of Technology. His research interests focus on the development of advanced materials for fuel cells, electrocatalytic and photocatalytic water splitting, and perovskite solar cells.



**Dr. Yufen Wang** obtained her Ph.D in 2013 from Sun Yat-sen University, then she worked at Tianjin Normal University. And she worked at Georgia Institute of Technology (2016–2017) as a visiting scholar. Her research interests include fabrications of hierarchically nanostructured materials and their applications in Solar Cells, catalysis and nanoscience.



**Dr. Bing Wang** received her Ph.D from Nanjing University in 2014. She then worked as an assistant research fellow at Nanjing University from 2015 to 2017, and a postdoctoral research associate in Prof. Zhiqun Lin's group in the School of Materials Science and Engineering at the Georgia Institute of Technology from 2017 to 2019. Her work involved synthesis of high-performance perovskite solar cells. Now she is an associate research fellow in the School of Physics at Nanjing University.